

GALAXI: Gallium anode low-angle x-ray instrument

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Abstract: The high brilliance laboratory small angle X-ray scattering instrument GALAXI, which is operated by JCNS, Forschungszentrum Jülich, permits the investigation of chemical correlations in bulk materials or of structures deposited on a surface at nanometre and mesoscopic length scales. The instrument is capable to perform GISAXS experiments in reflection at grazing incidence as well as SAXS experiments in transmission geometry. The X-ray flux on sample is comparable or higher than the one obtained at a comparable beamline at a second generation synchrotron radiation source.

1 Introduction

Small Angle X-ray Scattering (SAXS) permits the investigation of chemical correlations in bulk materials at nanometre and mesoscopic length scales. When the X-ray beam is sent under grazing incidence on a flat surface, reflectometry and Grazing Incidence Small Angle X-ray Scattering (GISAXS) can be performed, allowing the determination of the chemical depth-profile of multilayer thin films and the in-plane correlations at nanometre and mesoscopic length scales. The diffractometer of GALAXI is based on the JUSIFA anomalous SAXS instrument formerly installed at the beamline B1 of the DORIS storage ring at HASYLAB, DESY Hamburg, Germany. After the shutdown of DORIS, the instrument

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has been moved to the Research Centre of Jülich and has been reinstalled with a new X-ray source and a new position sensitive detector.

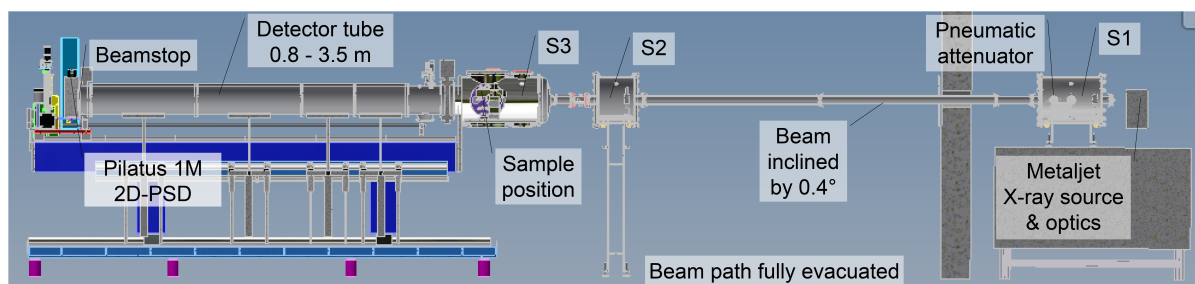


Figure 1: Schematic drawing of GALAXI with its main components. The beam direction is from right to left.

As X-ray source we use the METALJET source built by Bruker AXS. This source utilizes a liquid metal jet of a GaInSn alloy as anode. An electron beam of 20 μm height x 80 μm width at 70 keV energy and 200 W power hits the liquid metal jet and X-rays are produced. Parabolic Montel-type optics are used to parallelize the beam and monochromatize it by allowing only the Ga K_{α} radiation ($E = 9243$ eV photon energy) to pass. Due to the small source size, the intrinsic divergence of the X-ray beam is only 0.3 mrad at a maximum size of 2.4×2.4 mm².

After the optics, a collimation with two 4-segment slits separated by 4.0 m distance can be used to define the size and the collimation of the beam at the sample position. A third slit is used to reduce the background. At the sample position we receive a flux of $1 \cdot 10^9$ photons/mm²·s at 0.3 mrad divergence. At the sample position, the sample can be adjusted by two rotational and two translational degrees of freedom. A second sample holder contains several reference samples for calibration purposes. The detector distance can be adjusted between 835 mm and 3535 mm in 5 steps. The X-ray flight path is fully evacuated between the X-ray source and the detector. As detector, we use a Pilatus 1M 2D position sensitive detector with 169×179 mm² active area. The X-ray source is isolated from the diffractometer area in a separate room for radiation protection. The diffractometer room is freely accessible in all operation states.

All parts of GALAXI are controlled by a computer system according to the "Jülich-Munich" standard based on a Linux workstation. The computer system as well as the software are identical with the ones used at the JCNS outstation at MLZ Garching. This allows a flexible and efficient remote control with automatic scan programs.

2 Typical applications

2.1 Hard matter applications

A typical application of GALAXI is the determination of the size and size distribution of nanoparticles in solutions or deposited on surfaces as well as the ordering between those nanoparticles. Those nanoparticles are often magnetic for applications in information technology or spintronics (see Fig. 3). The diffractometer can also be used to measure reflectivity from layered thin films, in order to determine the thickness of the layers and the interfacial profiles.

2.2 Soft matter applications

The application range of GALAXI includes different kinds of polymer-based systems, in particular nanocomposite materials, polymers with different topology (ring, comb, branched polymers). Due to medium flux density, undesired radiation damage of biological substances is prevented. At the same time, high beam stability allows one to obtain reasonable quality data also for lower concentrations.

The instrument was successfully used for structural studies of proteins in crowded environment and also fragments of DNA.

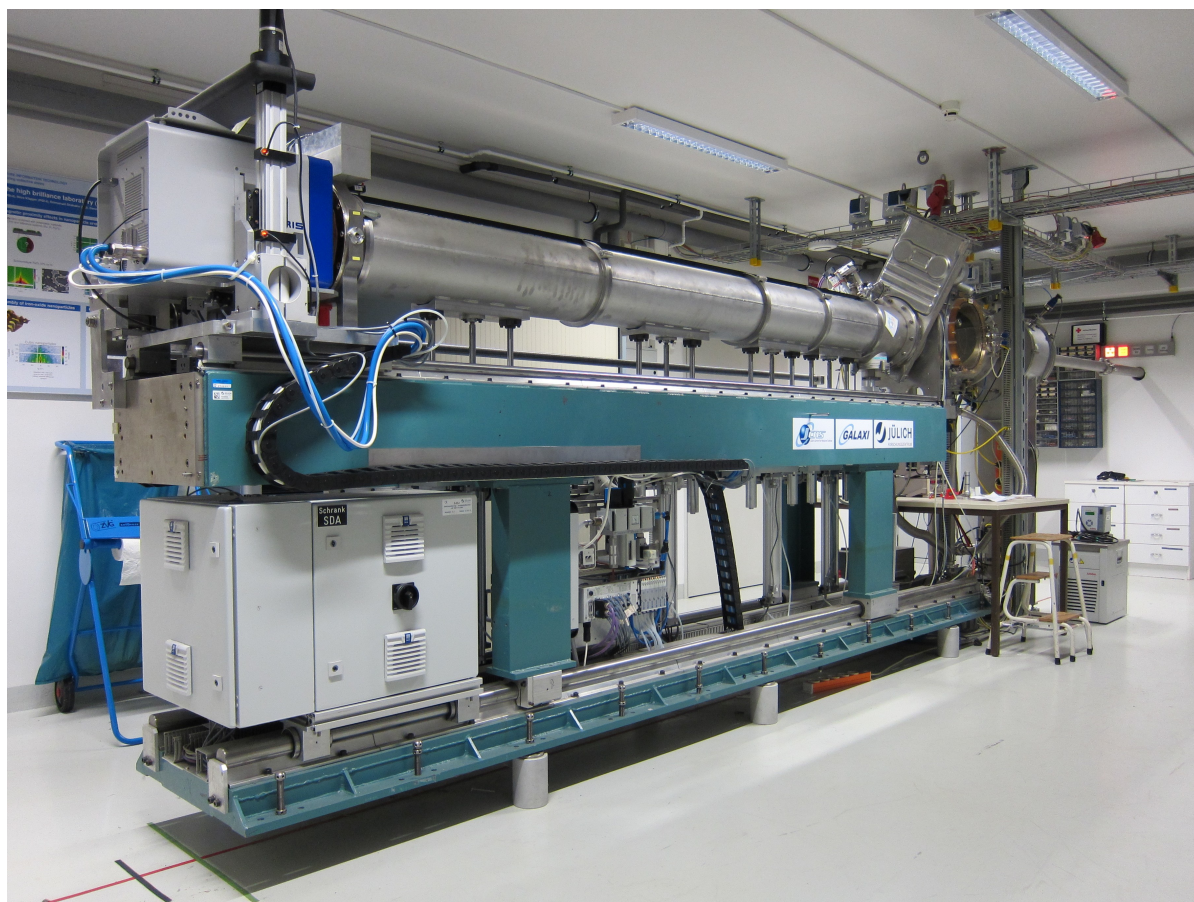


Figure 2: GALAXI diffractometer with the longest detector distance available. The X-ray source is located behind the wall, the detector is at the left hand side.

3 Sample environment

The samples are located in the main vacuum system of the x-ray flight path. Standard sample holders are available for sealed capillaries (for liquids and paste-like samples) with outer diameter from 1.5 to 2.1 mm, powders and thin film samples on substrates. In the case of transmission geometry, up to 11 samples can be mounted at the same time. For capillaries, we offer a heater / cooler setup that can be operated in the temperature range from 4 - 70°C.

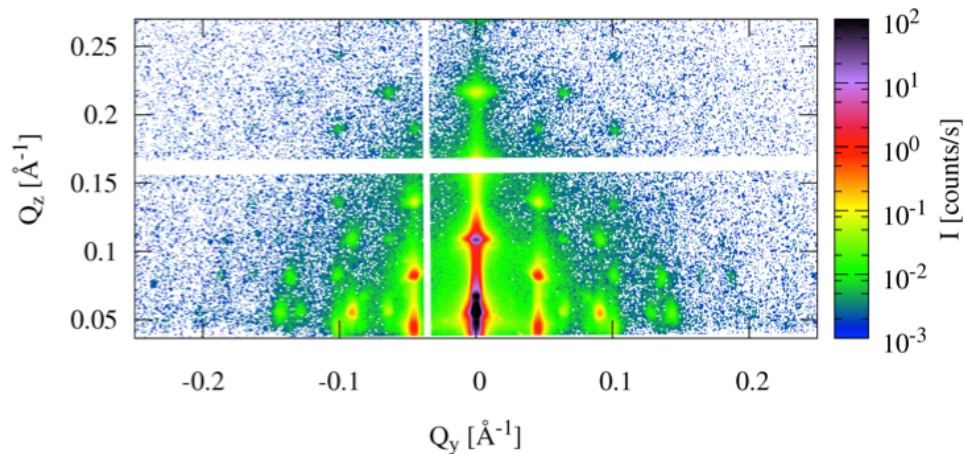


Figure 3: GISAXS measurement from an assembly of magnetic nanoparticles of cubic shape deposited on a substrate. From the positions of the peaks, one can deduce a body centred tetragonal ordering between nanoparticles. (Courtesy of E. Josten, Helmholtz-Zentrum Dresden-Rossendorf)

4 Technical data

4.1 X-ray source

- GaInSn liquid anode
- 200 W at 70 keV
- 20 μm height x 80 μm width of electron beam
- 20 μm diameter X-ray source projected to beam extraction direction
- Parabolic Montel type optics
- 2.4 x 2.4 mm^2 size of parallel beam
- 0.3 mrad intrinsic divergence
- Energy Ga K_{α} , $E = 9243 \text{ eV}$, $K_{\alpha 1}$ and $K_{\alpha 2}$ not resolved
- Wavelength $\lambda = 0.134 \text{ nm}$
- Beam inclined by 0.4° downwards

4.2 Collimation

- Two 4-segment slits (S1 and S2 in Fig. 1) at 0.3 m and 4.3 m distance from the optics
- Third 4-segment slit (S3) 0.9 m after S2 for reduction of background at small angles
- Pneumatic attenuator behind S1
- Fully evacuated collimation ($<1 \text{ mbar}$ pressure)

4.3 Sample position

- Located 1.1 m after S2
- Flux at sample position: $1 \cdot 10^9 \text{ photons/mm}^2 \cdot \text{s}$ at 0.3 mrad divergence
- Two translations perpendicular to the beam direction
- Two rotations with rotation axis perpendicular to the beam direction: in-plane rotation 360° , α_i up to 9.5°
- Reference sample holder with AgBH, Glassy Carbon and FEP reference samples at 14 cm behind the main sample position
- Sample position is evacuated

4.4 Detectors

4.4.1 Small-Angle detector

- Pilatus 1M 2D-position sensitive detector located in the small-angle scattering region
- Active area: 169 mm width x 179 mm height
- Distance between pixels: 0.172 mm horizontal and vertical
- Sample to detector distances available: 835 mm, 1285 mm, 1735 mm, 2635 mm, 3535 mm
- Evacuated detector tube
- Q-range: $4 \cdot 10^{-2} \dots 8 \text{ nm}^{-1}$

4.4.2 Single detectors and monitors

- Ionization chambers after optics and between S2 and S3
- PIN diodes after S1 and after sample position
- Calibrated PIN diode available at the sample position